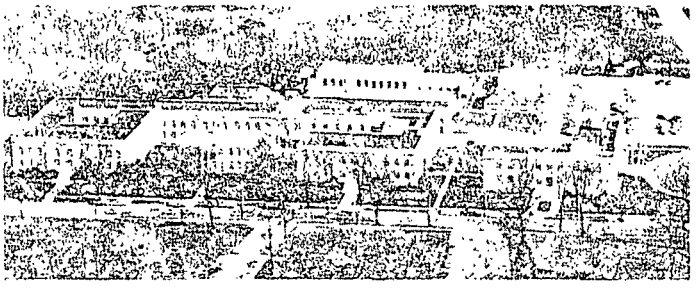

THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

DEVELOPMENT OF A MANUFACTURING PROCEDURE FOR LOW-LITHIUM, LOW-URANIUM CONTENT FILTER PAPER

Project 3101

Report Five

A Status Report

to

DEPARTMENT OF THE AIR FORCE
1155th TECHNICAL OPERATIONS SQUADRON (HQ. COMD.)
McCLELLAN AFB, CALIFORNIA

May 4, 1973

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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SUMMARY

By means of a series of three cartridges of ion-exchange resins, triple-deionized water was used in all process solutions and washing operations. The water contained approximately 0.0003 ng. U/ml. and 0.010 ng. Li/ml. with some samples < 0.007 ng. Li/ml.

A number of experiments with the cotton linters pulp (Hercules PS-57) and with IPC-1478 filter paper established that uranium levels well below the goal sought (< 0.3 ng./g.) can be obtained consistently by leaching the pulp or paper with hydrofluoric acid and/or ammonium carbonate.

The goal of < 0.4 ng. Li/g. of pulp or paper was obtained with three samples of the Hercules pulp, but the IPC-1478 filter paper and most samples of pulp contained lithium in amounts higher than the specified level. Work is in progress to try to exploit leaching and drying techniques which may lower the lithium contents as required.

Corrections: Strike the phrase "untreated with Kronisol" from the heading of Table III, Project 3101, Report Four, p. 8. Experiment 012-5541 used IPC-1478 paper which contained the usual amount of Kronisol. The paper in 012-5539 and 012-5540 contained no Kronisol.

DISCUSSION

PROCESS WATER

Based on previously reported data for the lithium content of the Institute's deionized water (1) (see 3101-026, -027, and -055), two cartridges of ion-exchange resins were placed in series with the ones already in use. As shown in Table I, the lithium content of the triple-deionized water averaged approximately 0.010 ng./ml., which is also the same as that of the triple-distilled water used by McClellan AFB, MCL. Thus, when water-to-pulp proportions greater than 40 ml./g. are used in pulp or paper purification, the lithium contents of the resultant pulp may be expected to hold substantially above the goal of < 0.4 ng./g. This assertion is based on the possibility that equilibria exist between lithium ions in the cellulose fibers and those in solution.

HERCULES COTTON LINTER PULP (PS-57)

Several modifications of the purification of pulp by leaching with hydrofluoric acid and ammonium carbonate were tried, and are summarized in Table II.

Each of the Samples 100, 101, and 114 involved the dispersion of 100 g. of pulp in 4 liters of solution. Even though the results were somewhat sporadic for both lithium and uranium, they showed that the addition of sodium ion failed to displace the lithium ion from sites in the cellulose, and that the addition of hydrochloric acid had little if any benefit.

TABLE II

HERCULES COTTON LINTER PULP (PS-57) LEACHED WITH HYDROFLUORIC ACID AND AMMONIUM CARBONATE[a]

Sample No.	Amount and Form of Pulp	Leaching Reagent[b]		Lithium, ng./g.	Uranium,[c] 8/5	Uranium,[c] ng./g.
3101-100	100 g. dispersed	4 liters 0.5M HF	A	1.35	128	0.310
			B	1.72	137	0.514
-101	100 g. dispersed	4 liters each of 0.5M HF, NaOH at 2.3 micrograms/g., 0.5M HF	A	2.64	137	0.466
			B	2.91	135	1.328
-104 (disks 4-7)	869 g., disks[d] (16 cm. in diameter)	4 liters 0.5M HF leached by percolation	A	0.547	117	0.159
			B	0.838	131	0.514
-107 (disks 34-37)			A	0.685	115	0.185
			B	0.524	115	0.183
-110 (disks 64-67)			A	0.486	122	0.215
			B	0.840	119	0.230
-113 (disks 94-97)			A	0.628	116	0.205
			B	0.531	117	0.211
-114	100 g. dispersed	4 liters of 2% of mixture of HCl:HF (2:1)	A	2.44	132	0.451
			B	0.812	125	0.183
-115 (Circles 3 and 4)	110 g. (five circles 26 cm. in diameter)	2 liters 0.5M HF containing 23 micrograms sodium ion/g. pulp; 1 liter 0.025M HF		0.667		
-116 (Circles 3 and 4)	110 g. (five circles 26 cm. in diameter)	2 liters 0.5M HF containing 23 mg. of sodium; 2 liters of HF at pH 3 containing 23 ng. sodium/g. pulp		1.003		
-117 (Circles 5-8)	57.5 g. (10 circles 12.5 cm. in diameter)	1 liter 0.5M HF containing 13.7 mg. sodium ion; 0.5 liter 0.025M HF		0.458	119	0.103
-118 (Circles 5-8)	57.5 g. (10 circles 12.5 cm. in diameter; Circles 5-8 processed between qual. filter paper)	1 liter 0.5M HF containing 13.7 mg. sodium ion; 0.5 liter 0.025M HF; 0.5 liter water		0.371		
-119 (top)	883 g. disks	2.0 liters 0.5M HF; 2.0 liters water; then half stack (440 g.) leached with 1.0 liter 0.5M HF		0.526		
-120 (middle)				0.532		
-121 (bottom)				0.553		

Samples 115 and 116 are consistent with 101 in showing that sodium ion may not displace lithium ion from the pulp. On the other hand, Samples 117 and 118 were processed in the presence of sodium ion and then dried between pulp circles (117) and qualitative filter paper circles (118). In the case of Sample 118 the lithium content was < 0.4 ng./g., the first among three samples (see 127 and 129) to reach the specified goal.

The remainder of the samples (119-131) represent various combinations of leaching pulp disks with hydrofluoric acid and ammonium carbonate with the analytical samples dried between adjacent disks to shield the samples from airborne impurities.

The use of lithium-6 in Samples 128-131 tended to confirm that some of the lithium apparently occupies sites where it may not be exchangeable with lithium ions in the process solutions. However, further work involving successive leaching and drying cycles, and increased time of leaching in hydrofluoric acid and ammonium carbonate would be necessary to establish the facts.

IPC-1478 FILTER PAPER

Based on the results obtained with the Hercules pulp (PS-57), samples of IPC-1478 paper (without Kronisol) were leached with hydrofluoric acid and ammonium carbonate, and with added sodium ions and lithium-6. The results are summarized in Table III. The results are consistent with previous results (1) and with those obtained for pulp. Samples 132-134 seem especially noteworthy because they resemble much more than previous samples the results obtained for the Hercules pulp PS-57. It may be noteworthy also that Samples 102 and 103 represent lot PM 7061A, Roll 1, Buckeye linters, double pass, and that Samples

132-134 represent lot PM 7062 B, Toll 1, SFP, single pass. The results suggest that the source of the linters from which the paper is made may be important in attaining the specifications for low lithium paper.

FUTURE WORK

1. Experiments are underway to test the possible usefulness of processing and drying IPC 1478 paper (no Kronisol added) between disks of pulp.

2. An experiment of limited scope is under way to test the effect, if any, of treating pulp with sodium borohydride before leaching with hydrofluoric acid.

3. Further results will be obtained with pulp and paper which have been repeatedly leached and dried.

3101-101 ATTEMPTED EXCHANGE OF SODIUM FOR LITHIUM IN PULP

An amount of 100 g. of pulp was leached with 0.5M hydrofluoric acid and washed with water as described in 3101-100 above. The wet pulp was then dispersed in 4 liters of sodium hydroxide containing 2300 ng. Na/g. of pulp, the mixture was allowed to stand for sixty minutes, was collected on the funnel without washing, and was pressed and dried overnight. The pulp was then leached with 0.5M hydrofluoric acid, washed with water, pressed, and dried as with 3101-100.

3101-102 LEACHING IPC-1478 PAPER WITH HYDROFLUORIC ACID

Three circles of IPC-1478 paper* (12.5 cm. in diameter) were placed on top one circle of pulp in a polyethylene funnel and three circles of pulp were placed on top the IPC-1478 paper. An amount of 1.0 liter of 0.5M hydrofluoric acid was percolated through the stack of circles, followed by 1.0 liter of water. The stack of circles was pressed under a rubber dam and the three paper circles were dried together in contact with the contacting pulp circle (third from top).

3101-103 LEACHING IPC-1478 PAPER WITH AMMONIUM CARBONATE (WITH SODIUM IONS) AND HYDROFLUORIC ACID

A stack of IPC-1478 paper and of pulp circles was arranged as in 3101-102, and was leached by percolation with 1.0 liter of a solution containing 5 g. of ammonium carbonate and 2.3 mg. of sodium ion (as hydroxide). The stack was then washed with 500 ml. of water followed by 1.0 liter of 0.5M hydrofluoric acid, and finally with 1.0 liter of water. The sample was air-dried as in 3101-102.

*All IPC-1478 paper described in this report was untreated with Kronisol.

through the stack of pulp. The stack was washed with 1 liter of 0.025M hydrofluoric acid followed by water until the effluent was neutral to Congo Red paper, pressed under a rubber dam, and dried in the usual way. The 3rd and 4th circles were selected as the sample for analysis.

3101-116

The experiment described under 3101-115 was repeated except that the pulp was leached with 2 liters of 0.5M hydrofluoric acid containing 25 mg. of sodium ion (110 ml. of 0.01N sodium hydroxide). Also, the pulp was washed with 1 liter of 0.025M hydrofluoric acid followed by 2 liters of hydrofluoric acid at pH 3 containing 23 ng. Na per gram of pulp.

3101-117 PULP (12.5 cm.) LEACHED WITH HYDROFLUORIC ACID AND SODIUM FLUORIDE

Ten circles, 12.5 cm. in diameter, of pulp were stacked in the small funnel and were leached by percolation with 1 liter of 0.5M hydrofluoric acid containing 12 mg. of sodium ion (55 ml. of 0.01N sodium hydroxide). The stack was then washed with 0.5 liter of 0.025M hydrofluoric acid followed by 0.5 liter of water, and pressed and dried. Circles 5-8 were submitted as a single sample for analysis.

3101-118

The above experiment was repeated except that circles of qualitative filter paper were placed above and below each pulp circle from 5 to 8. Thus, these pulp circles were dried between circles of qualitative filter paper to shield them from air-borne contamination.

The analytical samples were designated: upper, middle, and lower, 3101-125, -126, and -127, respectively.

3101-128 AND -129 PULP DISKS LEACHED WITH HYDROFLUORIC ACID CONTAINING LITHIUM-6

A stack of pulp disks, 400 g., was leached with 0.5 liter of 0.5M hydrofluoric acid containing 10 ng. ^{6}Li/g. pulp. This was followed by 0.5 liter of 0.5M hydrofluoric acid which contained no added lithium, and the stack was washed with 1.5 liters of water. The stack was pressed, and the disks were dried in sets of three with samples for analysis from the upper and lower sections of the stack.

3101-130 AND -131 PULP DISKS LEACHED WITH AMMONIUM CARBONATE CONTAINING LITHIUM-6

A stack of pulp disks, 400 g., was leached with 0.9 liter of 0.1M ammonium carbonate containing 19 ng. ^{6}Li/g. pulp. The stack was then leached with 0.5 liter of 0.1M ammonium carbonate containing no added lithium, followed by 1.5 liters of water, pressed, and dried in sets of three. Samples of the upper and lower sections of the stack were analyzed.

3101-132 IPC-1478 PAPER LEACHED WITH AMMONIUM CARBONATE AND HYDROFLUORIC ACID

Six circles, 12.5 cm. in diameter, of IPC-1478 paper (PM 7062B, Roll 1, SFP, single pass) were placed in sets of two between qualitative filter paper circles. These units were stacked on three circles of pulp and three circles of pulp were placed on top. The stack, total weight 49 g., was leached with 0.5 liter of 0.1M ammonium carbonate containing 11.5 micrograms of sodium ion

LITERATURE CITED

1. Project 3101, Report Four, March 6, 1973.

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